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Part III

Environmental Protection Agency

Lead-Acid Battery Manufacture; Proposed Standards of Performance and Notice of Public Hearing

ENVIRONMENTAL PROTECTION AGENCY

40 CFR Part 60

[FRL 1315-4]

Standards of Performance for New Stationary Sources; Lead-Acid Battery Manufacture

AGENCY: Environmental Protection Agency (EPA).

ACTION: Proposed rule and notice of public hearing.

SUMMARY: The proposed standards of performance would limit atmospheric emissions of lead from new, modified, and reconstructed facilities at lead-acid battery plants. This source was listed August 21, 1979 (44 FR 49222) in accordance with section 111(b)(1)(A) of the Clean Air Act as contributing significantly to air pollution, which may reasonably be anticipated to endanger public health or welfare. The intended effect of this proposal is to require new, modified, and reconstructed lead-acid battery manufacturing facilities to control lead emissions within the specified limits, which can be achieved through the use of the best demonstrated system of continuous emission reduction. A new reference method for determining compliance with lead standards is also proposed.

DATES: *Comments*—Comments must be received on or before March 14, 1980. *Public Hearing*—The public hearing will be held on Wednesday, February 13, 1980 beginning at 9:30 a.m. *Request to Speak at Hearing*—Persons wishing to present oral testimony must contact EPA by February 7, 1980.

ADDRESSES: *Comments*—Comments should be submitted (in duplicate if possible) to the Central Docket Section (A-130), U.S. Environmental Protection Agency, 401 M Street S.W., Washington, D.C. 20460, Attention: Docket No. OAQPS-79-1. *Public Hearing*—The public hearing will be held at EPA Environmental Research Center Auditorium, Rm B102, Research Triangle Park, N.C. 27711. Persons wishing to present oral testimony should notify Shirley Tabler, Emission Standards and Engineering Division (MD-13), Environmental Protection Agency, Research Triangle Park, North Carolina 27711, telephone number: (919) 541-5421. *Background Information Document*—The background information document for the proposed standards may be obtained from the U.S. EPA Library (MD-35), Research Triangle Park, North Carolina 27711, telephone number (919) 541-2777. Specify "Lead-Acid Battery Manufacture, Background Information for Proposed Emission Standards" (EPA 450/3-79-028a). *Docket*—The docket, number OAQPS-79-1, is available for public inspection and copying between 8:00 a.m. and 4:00 p.m. at EPA's Central Docket Section, Room 2903B, Waterside Mall, 401 M Street S.W., Washington, D.C. 20460.

FOR FURTHER INFORMATION CONTACT: Don R. Goodwin, Director, Emission Standards and Engineering Division (MD-13), Environmental Protection Agency, Research Triangle Park, North Carolina 27711, telephone number (919) 541-5271.

SUPPLEMENTARY INFORMATION:

Proposed Standards

The proposed standards would limit atmospheric lead emissions from new, modified, or reconstructed facilities at any lead-acid battery manufacturing plant which has a production capacity equal to or greater than 500 batteries per day (bpd). The facilities which would be affected by the standards, and the proposed emission limits for these facilities are listed below:

Facility:	*Lead emission limit*
Lead oxide production	5.0 mg/kg (0.010 lb/ton).
Grid casting	0.05 mg/m³ (0.2 x 10^{-4} gr/dscf).
Paste mixing	1.00 mg/m³ (4.4 x 10^{-4} gr/dscf).
Three-process	1.00 mg/m³ (4.4 x 10^{-4} gr/dscf).
Lead reclamation	2.00 mg/m³ (8.8 x 10^{-4} gr/dscf).
Other lead emitting operations.	1.00 mg/m³ (4.4 x 10^{-4} gr/dscf).

The emission limit for lead oxide manufacture is expressed in terms of lead emissions per kilogram of lead processed, while those for other facilities are expressed in terms of lead concentrations in exhaust air.

A standard of 0 percent opacity is proposed for emissions from any of these facilities. The proposed standards would also require continuous monitoring of the pressure drop across the control system for any affected facility, to help insure proper operation of the system. Performance tests would be required to determine compliance with the proposed standards. A new reference method, Method 12, would be used to measure the amount of lead in exhaust gases, and Method 9 would be used to measure opacity. Process monitoring would be required during all tests.

Summary of Environmental, Energy, and Economic Impacts

There are approximately 190 lead-acid battery manufacturing plants in the United States, of which about 100 have been estimated to have capacities greater than or equal to 500 batteries per day (bpd). These plants are scattered throughout the country and are generally located in urban areas near the market for their batteries. Projections of the growth rate of the lead-acid battery manufacturing industry range from 3 to 5 percent per year over the next 5 years. Most of the projected increase in manufacturing capacity is expected to take place by the expansion of large plants (producing over 2000 batteries per day).

New, modified, and reconstructed facilities coming on-line over the next 5 years will emit about 95 Mg (104 tons) of lead to the atmosphere in the fifth year, if their emissions are controlled only to the extent required by current State particulate regulations. At some existing plants, emissions are controlled to a greater extent than State particulate regulations require. This practice might be continued at new plants in the absence of the proposed standards of performance. The proposed standards would reduce potential lead emissions from facilities coming on-line during the next 5 years to about 2.8 Mg (3.1 tons) in the fifth year. This is approximately 97 percent lower than the emission level which would be allowed under State particulate regulations. The proposed standards would also result in decreased nonlead particulate emissions from new plants, since equipment installed for the purpose of controlling lead-bearing particulate emissions would also control nonlead-bearing particulate emissions.

The results of dispersion modeling calculations indicate that the ambient impact of lead emissions from a new plant complying with the proposed standards would be less than the national ambient air quality standard for lead of 1.5$\mu g/m^3$ (averaged over calendar quarter). This is an important consideration, since most lead-acid battery plants are located in urban areas. Results of EPA dispersion modeling calculations indicate that the ambient lead standard will not be met in the vicinities of plants controlling emissions only to the extent required by existing State regulations.

The impact of the proposed standards on the wastewater and solid waste emissions of a lead-acid battery plant would depend on the technique used by that plant to comply with the proposed standards. The best demonstrated system for reduction of lead emissions is the use of fabric filters. High energy impingement scrubbers could also be used, but would have higher energy requirements and operating costs than fabric filters. At plants using impingement scrubbing to control emissions, lead-bearing wastewater would be generated. This would be

treated along with other plant wastewater prior to being disposed from the plant. The fractional increase in the lead content of wastewater discharged from a plant using impingement scrubbing to control atmospheric lead emissions would be about 0.5 to 4.5 percent. At plants using fabric filtration to comply with the proposed standards, the captured pollutant would be reclaimed, and there would be no increase in wastewater or solid waste emissions due to the proposed standards.

The energy needed to operate control equipment required to meet the proposed standards at a new plant would be approximately 2 percent of the total energy needed to run the plant. The incremental energy demand resulting from the application of the proposed standards to the battery manufacturing facilities expected to come on-line over the next five years would be about 2.8 Gigawatt hours of electricity in the fifth year. Approximately 4.8 thousand barrels of oil would be required to generate this electricity.

The capital cost of the installed emission control equipment necessary to meet the proposed standards on all new facilities coming on-line nationwide during the first five years of the standards would be approximately $8.6 million. The total annualized cost of operating this equipment in the fifth year of the proposed standards would be about $4 million.

These costs and energy and environmental impacts are considered reasonable, and are not expected to prevent or hinder expansion of the lead-acid battery manufacturing industry. Economic analysis indicates that, for plants with capacities larger than or equal to 500 bpd, the costs attributable to the proposed standards could be passed on with little effect on sales. The average incremental cost associated with the proposed standard would be about 30¢ per battery. This is about 1.6 percent of the wholesale price of a battery.

Modification and Reconstruction of Existing Sources

In accordance with provisions applicable to all standards of performance, the proposed standards for the lead-acid battery manufacturing industry would apply to modified and reconstructed facilities, as well as to new facilities.

Under the modification provisions of 40 CFR 60.14, except in certain cases, an existing facility would be affected by the proposed standards if some physical or operational change is made to that facility which results in an increase in atmospheric lead emissions from that facility. Actions which would not be considered modifications, and thus would not cause an existing facility to become subject to the standards, regardless of any emission increase are:

1. Routine maintenance, repair, and replacement of components.

2. An increase in the production rate of a facility which is accomplished with an expenditure on the source containing the facility of less than 5.5 percent of the value of the facility. (This is the Internal Revenue Service annual asset guideline repair allowance for a facility at a lead-acid battery plant.)

3. An increase in the number of operating hours of a facility.

4. The use of an alternative raw material if the facility was designed to accommodate such material prior to this proposal.

5. The addition or use of any system or device whose primary function is the reduction of air pollutants, except when an emission control system is removed or is replaced by a system which the Administrator determines to be less environmentally beneficial.

6. The relocation or change in ownership of an existing facility.

Under the reconstruction provisions applicable to all standards of performance (40 CFR 60.15), an existing facility might become subject to the standards if its components were replaced to such an extent that the fixed capital cost of the new components exceeded 50 percent of the fixed capital cost that would be required to construct a comparable new facility. The standards would not apply, however, if the Administrator determined that it would not be technologically or economically feasible to meet the standards. Such determinations would be made on a case-by-case basis.

Rationale for the Proposed Standards—Selection of Source and Pollutant for Control

The manufacture of lead-acid batteries begins with the casting of lead grids and the production of lead oxide powder. The lead oxide powder is mixed with water and sulfuric acid to form a stiff paste, which is then pressed onto the lead grids. The pasted grids (plates) are cured, stacked, and connected (burned) into groups that form the individual elements of a lead-acid battery. The battery plates are converted to active electrodes by the formation process. In this process, the battery elements are immersed in dilute sulfuric acid, and direct current is passed between the plates to form active electrodes. Formation can take place either in an open acid bath (dry formation), or after the battery elements have been assembled into battery cases (wet formation). At some plants, scrap lead is recycled in a lead reclamation process.

Most of the operations discussed above are independent of one another. For instance, most small plants do not have lead oxide production or lead reclamation facilities, and some large companies sell lead oxide. However, stacking and burning of battery plates and assembly of elements into battery cases are generally accomplished in a single operation, called the three-process operation.

Most lead-acid battery plants are located near residential or urban areas. These plants emit lead-bearing particulate matter to the atmosphere. Sources of atmospheric lead emissions at lead-acid battery plants include lead oxide production, grid casting, paste mixing, three-process operation, and lead reclamation facilities. A National Ambient Air Quality Standard has been established for lead. The health and welfare effects of lead are presented in the document, "Air Quality Criteria for Lead" (EPA–600/8–77–017). Based on the results of EPA emission tests and the current required level of control, it is estimated that emissions from lead-acid battery plants could result in ambient lead concentrations which exceed the National Ambient Air Quality Standard. These emissions may reasonably be anticipated to endanger public health or welfare. Therefore, standards are proposed for lead emissions from lead-acid battery plants. Standards are not proposed for nonlead particulate emissions because such emissions are slight when compared with particulate emissions from other sources. Also, control of lead emissions would result in the reduction of other particulate emissions as well.

In addition to lead-bearing particulate matter, plants using dry formation techniques emit sulfuric acid mist. This mist results from the entrainment of sulfuric acid in hydrogen bubbles which are generated during the formation process. Wet formation usually takes place in covered battery cases. Therefore, acid mist emissions from wet formation are small.

Sulfuric acid mist has been previously determined to be a health related pollutant for the purposes of section 111(d) of the Clean Air Act. Therefore, the Administrator considered proposing standards for the lead-acid battery manufacturing industry which would limit acid mist emissions from the dry formation process. The setting of standards requiring control of acid mist emissions from new, modified, and

reconstructed formation facilities would require States to limit acid mist emissions from existing plants as well, under section 111(d).

EPA tests on dry formation at one plant indicate that the uncontrolled sulfuric acid mist emission rate from this facility is low (1.1 kg/1000 batteries). Dispersion modeling studies based on this emission rate indicate that the maximum ambient impact of sulfuric acid mist emissions from the dry formation process at a plant as large as 6500 batteries per day would be less than 1 $\mu g/m^3$ on an annual basis. Therefore, standards for acid mist are not being proposed at this time.

In contrast, two literature sources indicate that the acid mist emission rate from dry formation may be much higher (up to 19 k/1000 batteries) than the rate measured by EPA. However, EPA is unable to determine the reliability of the data contained in these sources because neither the test methods used nor the process operating conditions during testing are known. Therefore, although these sources indicate that acid mist emissions from the formation process may be significant, they do not provide a sufficient basis for an acid mist standard for the formation process.

It is of concern to the Administrator that the decision not to regulate acid mist emissions from formation is based on the results of tests at only one plant. The public is specifically invited to submit comments with supporting data on the issue of acid mist control. Based on the information received, EPA may take action regarding control of acid mist emissions from the formation process.

Applicability

The proposed standards of performance would apply to new, modified, or reconstructed facilities at any lead-acid battery plant that has the capacity to produce 500 or more batteries in a day (24 hours). Plants with capacities less than 500 bpd are exempted from the proposed standards for several reasons. First, projections of the economic impact of standards on existing small plants (100 and 250 bpd) undergoing reconstruction or modification indicated that standards would have a severe negative impact on such plants. Also, although almost 50 percent of the lead-acid battery plants in the United States produce fewer than 500 bpd, these plants account for only about 2 percent of total lead-acid battery production. Finally, industry representatives do not forecast construction or expansion of small plants. In fact there has been a trend in recent years of small plants closing due to unprofitability. Increased demand for batteries in the future is expected to be accommodated by expansion of existing plants producing over 2000 bpd.

Selection of Affected Facilities

Lead emitting process operations selected as affected facilities are lead oxide production, grid casting, paste mixing, three-process operation, lead reclamation, and other lead emitting operations. These process operations often consist of several machines or production lines which perform the same function and which are located in the same area and ducted to the same control device. Therefore, for each of the process operations mentioned above, the affected facility is the entire operation. For example, at a plant with more than one three-process line, all of the lines together would be the affected facility.

Lead Oxide Manufacturing—In the lead-acid battery industry, lead oxide is produced either by the ball mill process, or by the Barton process. In the ball mill process, which is used more frequently, lead pigs or balls are tumbled in a mill, and the frictional heat generated by the tumbling action causes the formation of lead oxide. The lead oxide is removed from the mill by an air stream. In the Barton process, molten lead is atomized to form small droplets in an air stream. These droplets are then oxidized by the air around them.

Thus, in both the Barton process and the ball mill process, lead oxide product must be recovered from an air stream. In both processes, fabric filtration is used to accomplish this separation. It is estimated that lead emissions from a typical lead oxide manufacturing facility including product recovery are about 0.05 kg (o.116 lb) per 1,000 batteries, or about 10 g/Mg (0.02 lb/ton) of lead processed. Although these lead emissions are low, source tests have indicated that a well controlled lead oxide manufacturing facility would emit only about half as much lead as one designed only for economical recovery of lead oxide. Therefore, the lead oxide production process is designated an affected facility.

Grid Casting—Although lead emissions from grid casting are generally low, most grid casting work areas would be ventilated to comply with the in-plant OSHA lead concentration stanard of 50 $\mu g/m^3$. EPA tests detected an uncontrolled lead emission rate of 0.4 kg (0.9 lb) per 1,000 batteries (4.37 mg/m^3 or 19.1×10^{-4} gr/dscf of exhaust air), which was about 3.2 percent of the overall plant uncontrolled lead emission rate. Therefore, grid casting is designated an affected facility. The grid casting facility is defined to include both the grid casting machines, and the lead melting pots associated with these machines.

Paste Mixing—Paste mixing is generally a batch operation consisting of two phases: a charging phase, in which lead oxide is fed to the paste mixer, and a mixing phase, in which the lead oxide is mixed with water and sulfuric acid. Most emissions from paste mixing occur during the charging phase. However, lead emissions are also ducted from the paste mixer during the mixing phase. Uncontrolled lead emissions from paste mixing have been determined to be approximately 5.1 kg (11.2 lb) per 1,000 batteries (77.4 mg/m^3 or 338×10^{-4} gr/dscf of exhaust). This is about 40 percent of the total estimated uncontrolled lead emission rate for a lead-acid battery plant. The paste mixing facility is, therefore, selected as an affected facility.

Three-process Operation—The Three-process operation facility is defined to include all processes involved with plate stacking, burning, and assembly of elements into battery cases. Average uncontrolled lead emissions from three-process operations tested by EPA were 6.7 kg (14.7 lb) per 1,000 batteries (28 mg/m^3 or 115×10^{-4} gr/dscf of exhaust air). This is over 50 percent of the estimated lead emissions from a lead-acid battery plant. Therfore, the three-process operation is designated an affected facility.

Lead Reclamation—Lead reclamation is often a sporadic operation, on stream only when large quantities of defective small parts, grids, and plates are available. EPA measured lead emissions from this process to be 0.35 kg (0.77 lb) per 1000 batteries, 3.0 kg/Mg (5.9 lb/ton) of lead charged, or 227 mg/m^3 (990×10^{-4} gr/dscf) of exhaust air. Lead emissions can be very high during operation. For example, a 4000-bpd plant at which the lead reclamation facility is run for an 8-hour shift every 2 weeks would emit approximately 1.7 kg/hr (3.8 lb/hr) during operation. This is comparable with the lead emission rate from the three-process operation at the same plant. Therefore, the Administrator designates lead reclamation as an affected facility. Reverberatory furnances which are used for lead reclamation but which are affected by standards of performance for secondary lead smelters (40 CFR 60.120), would not be affected under the proposed standards.

Other Lead-Emitting Operations—Any lead-acid battery plant facility from which lead emissions are collected and ducted to the atmosphere but which is not considered part of the lead oxide

production, grid casting, paste mixing, three-process operation, or lead reclamation facilities is considered an "other lead emitting operation." An example is slitting, a process whereby lead grids, cast in doublets, are slit (with an enclosed saw) into separate plates. The Administrator has selected other lead emitting operations as affected facilities to ensure the control of lead emissions from these processes.

Selection of the Best System of Emissions Reduction Considering Costs

Section 111 of the Clean Air Act requires that standards of performance reflect the degree of emission control achievable through application of the best demonstrated technological system of continuous emission reduction which (taking into consideration the cost of achieving such emission reduction, any nonair quality health and environmental impact, and energy requirements) has been adequately demonstrated. The proposed standards were developed based on information derived from (1) available technical literature on the lead-acid battery manufacturing industry and applicable emission control technology, (2) technical studies performed for EPA by independent research organizations, (3) information obtained from the industry during visits to lead battery plants and meetings with various representatives of the industry, (4) comments and suggestions solicited from experts, and (5) results of emission measurements conducted by EPA.

Techniques currently used to control atmospheric lead emissions from facilities at lead-acid battery plants include fabric filtration and impingement scrubbing of exhaust gases. Low energy scubbers are currently used to control emissions entrained in hot gases, or in gases containing moisture or possible spark hazards. Fabric filters can be used to control all atmospheric lead emissions from lead-acid battery manufacturing, provided that proper maintenance procedures are followed and that necessary precautions are taken to prevent condensation or sparks when necessary. They are commonly used in other industries to control emissions from sources having similar moisure problems and spark problems.

Several emission control alternatives were studied in the development of the proposed standards. One of these alternatives consists of fabric filter control for all affected facilities. The others consist of fabric filter control for some affected facilities and low energy impingement scrubber control for other affected facilities. The alternative which would achieve the best degree of emission control is fabric filter control of atmospheric lead emissions from all affected facilities. The other alternatives make use of low energy scrubbers to varying extents, and represent varying degrees of emission reduction. Economic analysis has shown that, for plants with capacities greater than or equal to 500 batteries per day, none of the control alternatives which were studied would impose an unreasonable cost. Also, the cost of any alternative is not viewed as being detrimental to industry expansion.

The proposed standards are based on the control of all lead emissions from lead-acid battery plants by fabric filtration. This basis was chosen because fabric filters can achieve a better degree of emission reduction than low energy scrubbers at a reasonable cost.

The use of control techniques other than fabric filtration would not be precluded by the proposed standards. High energy impingement scrubbers could be used to meet the emission limits. However, these would have higher operating costs and energy requirements than fabric filters. Scrubbers would also generate lead contaminated water, which would probably require treatment prior to disposal.

Selection of the Format for the Proposed Standard

In general, lead-acid battery manufacturing facilities may be considered independent of one another in that there is no continuous flow of materials. Lead oxide production operations, grid casting operations, paste mixing operations, lead reclamation operations, and three-process operations are independent. Also, not all plants have lead reclamation and lead oxide production operations, and some plants sell lead oxide.

Because of the independent nature of the facilities, two different forms were chosen for the proposed standards. The format of the proposed standards applicable to grid casting, paste mixing, three-process operation, lead reclamation, and other lead-emitting operations, is a concentration standard. The format of the standard for lead oxide manufacturing is mass per unit of lead input.

A concentration standard is proposed for three-process facilities because emissions from these facilities depend more on number of plates processed and the method of burning than on the weight of material processed. Since emissions from grid casting, paste mixing, and lead reclamation are often controlled by the control device which controls three-process operation emissions, concentration standards are also proposed for these facilities. A mass per unit lead input standard is proposed for lead oxide production facilities because these facilities generally have different emission control devices from three-process operation facilities and because emissions from lead oxide production are generally proportional to lead input.

Selection of Emission Limits

The proposed limits for lead emissions from lead oxide production, grid casting, paste mixing, three-process operation, lead reclamation, and other lead emitting facilities are based on emissions levels attainable using fabric filtration. In the development of background data for the proposed standards, atmospheric lead emissions from facilities at four lead-acid battery plants were measured using the proposed Method 12. In a previous study, lead emissions from facilities at two lead-acid battery manufacturing plants and one lead oxide manufacturing plant were measured using a similar test method.

The emission limits for three-process operation facilities, lead oxide production facilities, and other lead emitting facilities are based on lead levels measured in exhausts from fabric filters controlling emissions from such facilities. Fabric filters are not currently used in the lead-acid battery industry to control emissions from grid casting or lead reclamation and are not generally used to control emissions from the mixing phase of paste mixing. The emission limits for grid casting, paste mixing, and lead reclamation are, therefore, based on lead levels found in uncontrolled emissions from such facilities, and on the demonstrated emission reduction capabilities of fabric filters.

Tests of controlled and uncontrolled emissions from three-process facilities controlled by fabric filters indicated fabric filter lead collection efficiencies of about 99 percent. This control efficiency is consistent with efficiencies achieved by well maintained fabric filters in other applications. Because particulate emissions from all lead emitting facilities at battery plants are similar in composition and particle size, the Administrator has determined that comparable collection efficiencies can be achieved for emissions from grid casting, paste mixing, and lead reclamation.

Lead Oxide Manufacturing—The proposed standard for lead oxide production is 5 milligrams of lead per kilogram of lead processed (10 lb/ton).

This limit is based on results of tests of emissions from a ball mill lead oxide production facility with a fabric filter control system. The tests showed an average controlled emission rate of 4.2 mg/Kg (8.4 lb/ton) for this facility. EPA has not conducted tests of emissions from a well controlled Barton process. However, in both the ball mill process and the Barton process, lead oxide product must be removed from an air stream. Also, EPA tests on a Barton process indicated that Barton and ball mill processes have similar air flow rates per unit production rate. Therefore, it has been determined that a similar level of emission control could be achieved for a Barton process as has been demonstrated for the ball mill process.

Grid Casting—Impingement scrubbing, rather than fabric filtration, is currently used in the lead-acid battery manufacturing industry to control emissions from grid casting. Emissions from grid casting facilities were measured at two plants. At one of these plants, grid casting emissions were controlled by an impingement scrubber. At the other, grid casting emissions were not controlled. The average lead concentration in exhaust from the uncontrolled facility was 4.37 mg/m^3 (19.1×10^{-4} gr/dscf). Average uncontrolled and controlled lead emissions from the scrubber controlled facility were 2.65 mg/m^3 (11.6×10^{-4} gr/dscf) and 0.32 mg/m^3 (1.4×10^{-4} gr/dscf), respectively. Thus the lead collection efficiency of the scrubber was about 90 percent.

Fabric filtration can be used to control these emissions if spark arresters are used and the exhaust gas is kept above the dew point. The lead standard for grid casting, 0.05 mg/m^3 (0.2×10^{-4} gr/dscf), is based on the exhaust concentration achievable using a fabric filter with about 99 percent collection efficiency to control emissions.

Paste Mixing—Lead emissions from a paste mixing facility equipped with an impingement scrubber were measured. Average uncontrolled and controlled lead concentrations from this facility were 77.4 mg/m^3 (338×10^{-4} gr/dscf) and 10.8 mg/m^3 (47.0×10^{-4} gr/dscf), respectively.

Fabric filtration is not generally used to control emissions from the entire paste mixing cycle because of the high moisture content of paste mixer exhaust during the mixing cycle. However, fabric filtration can be used to control emissions from the entire cycle if the exhaust gas is kept above the dew point. The proposed lead emission standard for paste mixing, 1 mg/m^3 (4.4×10^{-4} gr/dscf), is based on the level achievable using a fabric filter with about 99 percent collection efficiency for the entire cycle.

In developing data for the proposed standards, EPA conducted tests at a plant where paste mixing emissions were controlled by two separate systems. At this plant, paste mixing required a total of 21 to 24 minutes per batch. During the first 14 to 16 minutes of a cycle (the charging phase), exhaust from the paste mixer was ducted to a fabric filter which also controlled emissions from the grid slitting (separating) operation. During the remainder of the cycle (mixing), paste mixer exhaust was ducted to an impingement scrubber which also controlled emissions from the grid casting operation. Uncontrolled or controlled emissions for the paste mixer alone were not tested. The average concentration of lead in emissions from the fabric filtration system used to control charging emissions was 1.3 mg/m^3 (5.5×10^{-4} gr/dscf). The average lead content of exhaust from the scrubber used to control mixing emissions was 0.25 mg/m^3 (1.1×10^{-4} gr/dscf). The average lead concentration in controlled emissions from this facility was about 0.95 mg/m^3 (4.2×10^{-4} gr/dscf) which is slightly below the proposed emission limit of 1 mg/m^3 (4.4×10^{-4} gr/dscf). A lower average emission concentration could be achieved by using fabric filtration to control emissions from all phases of paste mixing.

Three-Process Operation—The proposed lead concentration limit for three-process operation emissions is 1 mg/m^3 (4.4×10^{-4} gr/dscf). This limit is based on the results of EPA tests conducted at four plants where fabric filtration was used to control three-process operation emissions. All of these tests showed lead concentration below the proposed limit in controlled emissions from the three-process operation facilities.

Lead Reclamation—Lead emissions from a lead reclamation facility where emissions are controlled by an impingement scrubber were measured. The average lead concentrations in the inlet and outlet streams of the scrubber were 227 mg/m^3 (990×10^{-4} gr/dscf) and 3.7 mg/m^3 (16×10^{-4} gr/dscf), respectively. The collection efficiency of the scrubber was, therefore, about 98 percent.

Fabric filtration is not currently used to control emissions from lead reclamation facilities because of the high temperature of lead reclamation exhaust. However, fabric filters have been applied to hot exhaust systems at secondary lead smelters and in other industries. Therefore, the proposed standard for lead reclamation facilities of 2 mg/m^3 (8.8×10^{-4} gr/dscf), is based on the emission level attainable using a fabric filter with a collection efficiency of about 99 percent.

Other Lead Emitting Operations—Emissions from other lead emitting operations are generally collected and ducted to minimize worker exposure. These emissions are similar in composition and concentration to emissions from non-automated three-process operations. The proposed standard for other lead emitting operations is 1 mg/m^3 (4.4×10^{-4} gr/dscf) because lead emissions from those operations can be controlled to the same extent as lead emissions three-process operation facilities.

EPA measured emissions from a slitting facility, which would be classified as an "other lead emitting operation," controlled by a fabric filter. The controlled emissions from the facility had an average lead content of 0.938 mg/m^3 (4.1×10^{-4} gr/dscf), which is below the proposed concentration limit for other lead emitting operations.

Opacity Standards

A standard of 0 percent opacity is proposed for emissions from all affected facilities. Grid casting, paste mixing, three-process operation, and lead oxide manufacturing facilities were observed by EPA to have emissions with 0 percent opacity during observation periods of 7 hours and 16 minutes, 1 hour and 30 minutes, 3 hours and 51 minutes, and 3 hours and 19 minutes, respectively. Emissions ranging from 5 to 20 percent opacity were observed for a total of 11 minutes and 15 seconds during 3 hours and 22 minutes of observation at the lead reclamation operation source tested by EPA, which was controlled by a low-energy scrubber. However, the proposed standard is based on control of this process by a fabric filter, similar to three-process operations and paste mixers for which emissions with 0 percent opacity have been observed. A standard of 0 percent opacity is, therefore, also proposed for emissions from lead reclamation furnaces.

Under the proposed standards, opacity would be determined by taking the average opacity over a 6-minute period using EPA Test Method 9, and rounding the average to the nearest whole percentage. The rounding procedure is specified in the proposed standards in order to allow occasional brief emissions with opacities greater than 0 percent. When a fabric filter is used to control emissions, the outlet concentration from the filter may increase immediately after a component filter bag is cleaned. In the case of a

lead-acid battery plant, filter cleaning may result in occasional emissions with opacities greater than 0 percent. If the rounding off procedure were not specified, any reading of greater than 0 percent opacity during a 6-minute period could be considered as indicative of a violation of the proposed 0 percent opacity standard. However, the Administrator does not intend for occasional emissions greater than 0 percent opacity occurring during filter cleaning to be considered violations of the proposed standards. Therefore, the standards would specify that the average opacity be rounded to the nearest whole percentage. With this specification, 6-minute average opacities less than 0.5 percent would not be considered violations of the proposed standards. Emissions which result in 6-minute average opacities of 0.5 percent or greater are expected to be indicative of fabric filter malfunctions rather than filter cleaning emissions.

Testing and Recordkeeping

Performance tests would be required to determine compliance with the proposed standards. A new Reference Method 12 would be used to measure lead emissions. In addition, the following methods would be used to determine the necessary emission data: Method 1 for sample and velocity traverses, Method 2 for velocity and volumetric flow rate, Method 4 for stack gas moisture and Method 9 for stack opacity.

A measurement of the mass rate of feed would also be required during performance tests for lead oxide manufacturing because the units of the standards for this facility are milligrams of lead per kilogram of lead feed. Lead ingots of constant weight can be counted as they are fed to the lead oxide manufacturing process. The mass rate of feed measurements must be accurate within ±5 percent.

To determine compliance when two or more facilities at the same plant are ducted to a common control device, the exhaust rate from each source and the controlled lead concentrations must be measured. An equivalent standard for the applicable facilities is calculated by multiplying each applicable standard by the fractional exhaust flow rate of that facility and adding the numbers. This equivalent standard can then be compared with the measured concentration to determine compliance.

The proposed standards would require continuous monitoring of the pressure drop across the fabric filter or scrubber as applicable. A decrease in pressure drop of about 50 percent could indicate a decrease in lead removal efficiency because of either a fabric filter bag failure or a decrease in liquid-to-gas ratio.

EPA Reference Method 12 was developed to determine inorganic lead emissions from stationary sources. Particulate and gaseous lead emissions are withdrawn isokinetically from the source. The collected samples are then digested in acid solution and analyzed by atomic absorption spectrometry using an air acetylene flame. For a minimum analysis accuracy of ±10 percent, a minimum lead mass of 10μg should be collected. The typical sensitivities for a 1 percent change in absorption (0.0044 absorbance units) are 0.2 and 0.5 μg Pb/ml for the 217.0 and 283.3 nm lines, respectively.

The laboratory precision for Method 12, as measured by the coefficient of variation, was determined at a gray iron foundry, a lead battery manufacturing plant, a secondary lead smelter, and a lead recovery furnace at an alkyl lead manufacturing plant. The concentrations encountered during these tests ranged from 0.61 to 123.3 $\mu g/Pb/m^3$. The coefficient of variation for each run, which is the standard deviation of the run expressed as a percentage of the run mean concentration, ranged from 0.2 to 9.5 percent.

High copper concentrations may interfere with the analysis of lead at 217.0 nm. This interference can be avoided by analyzing the samples for lead using the 283.3 nm lead line. This problem should not occur, however, when analyzing samples from lead-acid battery facilities.

Records of performance tests and continuous monitoring system measurements would have to be retained for at least 2 years following the date of the measurements by owners and operators subject to this subpart. This requirement is included under § 60.7(d) of the general provisions of Part 60.

Public Hearing

A public hearing will be held to discuss these proposed standards in accordance with Section 307(d)(5) of the Clean Air Act. Persons wishing to make oral presentations should contact EPA at the address given in the ADDRESSES Section of this preamble. Oral presentations will be limited to 15 minutes each. Any member of the public may file a written statement with EPA before, during, or within 30 days after the hearing. Written statements should be addressed to the Central Docket Section (A–130), U.S. Environmental Protection Agency, 401 M Street, S.W., Washington, D.C. 20460, Attention: Docket No. OAQPS–79–1.

A verbatim transcript of the hearing and written statements will be available for public inspection and copying during normal working hours at EPA's Central Docket Section, Room 2903B, Waterside Mall, 401 M Street, S.W., Washington, D.C. 20460.

Docket

The docket, containing all supporting information used by EPA to date, is available for inspection and copying between 8:00 a.m. and 4:00 p.m., Monday through Friday, at EPA's Central Docket Section, room 2903B, Waterside Mall, 401 M Street, S.W., Washington, D.C. 20460.

The docket is an organized and complete file of all the information submitted to or otherwise considered by EPA in the development of the rulemaking. The docket is a dynamic file, since material is added throughout the rulemaking development.

The docketing system is intended to allow members of the public and industries involved to readily identify and locate documents so that they can intelligently and effectively participate in the rulemaking process. On judicial review, the record will consist of all materials in the docket except for certain interagency review materials (section 307(d)(7)(A) of the Act).

Miscellaneous

In accordance with section 117 of the Act, publication of these proposed standards was preceded by consultation with appropriate advisory committees, independent experts, and Federal departments and agencies. The Administrator will welcome comments on all aspects of the proposed regulation, including economic and technological issues, and on the proposed test method.

It should be noted that standards of performance for new sources established under section 111 of the Clean Air Act reflect:

> * * * application of the best adequately demonstrated technological system of continuous emission reduction which (taking into consideration the cost of achieving such emission reduction, any nonair quality health and environmental impact, and energy requirements) the Administrator determines has been adequately demonstrated [section 111(a)(1)].

Although there may be emission control technology available that can reduce emissions below those levels required to comply with standards of performance, this technology might not be selected as the basis of standards of performance because of costs associated with its use. Accordingly, standards of performance should not be

viewed as the ultimate in achievable emission control. In fact, the Act requires (or has the potential for requiring) the imposition of a more stringent emission standard in several situations.

For example, applicable costs do not necessarily play as prominent a role in determining the "lowest achievable emissions rate" for new or modified sources located in nonattainment areas, i.e., those areas where statutorily mandated health and welfare standards are being violated.

In this respect, section 173 of the Act requires that a new or modified source constructed in an area that exceeds the National Ambient Air Quality Standards (NAAQS) must reduce emissions to the level which reflects the "lowest achievable emission rate" (LAER), as defined in section 171(3), for such category of source. The statute defines LAER as that rate of emissions based on the following, whichever is more stringent:

(A) the most stringent emission limitation which is contained in the implementation plan of any State for such class or category of source, unless the owner or operator of the proposed source demonstrates that such limitations are not achievable, or

(B) the most stringent emission limitation which is achieved in practice by such class or category of source.

In no event can the emission rate exceed any applicable new source performance standard [section 171(3)].

A similar situation may arise under the prevention of significant deterioration of air quality provisions of the Act (Part C). These provisions require that certain sources [referred to in section 169(1)] employ "best available control technology" [as defined in section 169(3)] for all pollutants regulated under the Act. Best available control technology (BACT) must be determined on a case-by-case basis, taking energy, environmental and economic impacts, and other costs into account. In no event may the application of BACT result in emissions of any pollutants that will exceed the emissions allowed by any applicable standard established pursuant to section 111 (or 112) of the Act.

In all events, State Implementation Plans (SIP's) approved or promulgated under section 110 of the Act must provide for the attainment and maintenance of NAAQS designed to protect public health and welfare. For this purpose, SIP's must in some cases required greater emission reductions than those require by standards of performance of new sources.

Finally, States are free under section 116 of the Act to establish even more stringent emission limits than those established under section 111 or those necessary to attain or maintain the NAAQS under section 110. Accordingly, new sources may in some cases be subject to limitations more stringent than EPA's standards of performance under section 111, and prospective owners and operators of new sources should be aware of this possibility in planning for such facilities.

EPA will review this regulation 4 years from the date of promulgation. This review will include an assessment of such factors as the need for integration with other programs, the existence of alternative methods, enforceability, and improvements in emission control technology.

Section 317 of the Clean Air Act requires the Administrator to prepare an economic impact assessment for any new source standard of performance promulgated under section 111(b) of the Act. An economic impact assessment was prepared for the proposed regulations and for other regulatory alternatives. All aspects of the assessment were considered in the formulation of the proposed standards to insure that the proposed standards would represent the best system of emission reduction considering costs. The economic impact assessment is included in the background information document.

Dated: December 19, 1979.

Douglas M. Costle,

Administrator.

It is proposed that 40 CFR Part 60 be amended by adding a new Subpart KK and by adding a new reference method to Appendix A as follows:

1. A new subpart KK is added as follows:

Subpart KK—Standards of Performance for Lead-Acid Battery Manufacturing Plants

Sec.

60.370 Applicability and designation of affected facility.
60.371 Definitions.
60.372 Standards for lead.
60.373 Monitoring of emissions and operations.
60.374 Test methods and procedures.

Authority: Sec. 111, 301(a), Clean Air Act as amended [42 U.S.C. 7411, 7601(a)], and additional authority as noted below.

Subpart KK—Standards of Performance for Lead-Acid Battery Manufacturing Plants

§ 60.370 Applicability and designation of affected facility.

(a) The provisions of this subpart are applicable to the affected facilities listed in paragraph (b) of this section at any lead-acid battery manufacturing plant that has the capacity to produce 500 or more batteries per day (24 hours).

(b) The provisions of this subpart are applicable to the following affected facilities used in the manufacture of lead-acid storage batteries:

(1) Grid casting facility
(2) Paste mixing facility
(3) Three-process operation facility
(4) Lead oxide manufacturing facility
(5) Lead reclamation facility
(6) Other lead-emitting operations

§ 60.371 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in Subpart A of this part.

(a) "Grid casting facility" means the facility which includes both lead melting pots and machines used for casting the grids used in battery manufacturing.

(b) "Lead-acid battery manufacturing plant" means any plant that produces a storage battery using lead and lead compounds for the plates and sulfuric acid for the electrolyte.

(c) "Lead oxide manufacturing facility" means the facility that produces lead oxide from lead, including product recovery.

(d) "Lead reclamation facility" means the facility that remelts lead scrap and casts it into lead ingots for use in the battery manufacturing process, and which is not a furnace affected under Subpart L of this part.

(e) "Other lead-emitting operation" means any lead-acid battery manufacturing plant operation from which lead emissions are collected and ducted to the atmosphere and which is not part of a grid casting, lead oxide manufacturing, lead reclamation, paste mixing, or three-process operation facility, or a furnace affected under Subpart L of this part.

(f) "Paste mixing facility" means the facility including charging and blending of the ingredients to produce a lead oxide paste.

(g) "Three-process operation facility" means the facility including those processes involved with plate stacking, burning or strap casting, and assembly of elements into the battery case.

§ 60.372 Standards for lead.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere:

(1) From any grid casting facility any gases that contain lead in excess of 0.05 milligram of lead per dry standard cubic meter of exhaust (0.00002 gr/dscf).

(2) From any paste mixing facility any gases that contain in excess of 1.00 milligram of lead per dry standard cubic meter of exhaust (0.00044 gr/dscf).

(3) From any three-process operation facility any gases that contain in excess of 1.00 milligram of lead per dry standard cubic meter of exhaust (0.00044 gr/dscf).

(4) From any lead oxide manufacturing facility any gases that contain in excess of 5.0 milligrams of lead per kilogram of lead feed (0.010 lb/ton).

(5) From any lead reclamation facility any gases that contain in excess of 2.00 milligrams of lead per dry standard cubic meter of exhaust (0.00088 gr/dscf).

(6) From any other lead-emitting operation any gases that contain in excess of 1.00 milligram per dry standard cubic meter of exhaust (0.00044 gr/dscf).

(7) From any affected facility any gases with greater than 0 percent opacity (measured according to Method 9 and rounded to the nearest whole percentage).

(b) When two or more facilities at the same plant (except the lead oxide manufacturing facility) are ducted to a common control device, and equivalent standard for the total exhaust from the commonly controlled facilities shall be determined as follows:

$$S_e = \sum_{a=1}^{N} S_a(Q_{sd_a}/Q_{sd_T})$$

Where:

S_e is the equivalent standard for the total exhaust stream.

S_a is the actual standard for each exhaust stream ducted to the control device.

N is the total number of exhaust streams ducted to the control device.

Q_{sd_a} is the dry standard volumetric flow rate of the effluent gas stream from each facility ducted to the control device.

Q_{sd_T} is the total dry standard volumetric flow rate of all effluent gas streams ducted to the control device.

§ 60.373 Monitoring of emissions and operations.

The owner or operator of any lead-acid battery manufacturing plant subject to the provisions of this subpart shall install, calibrate, maintain, and operate a monitoring device(s) that continuously measures and premanently records the total pressure drop across the process emissions control system. The monitoring device shall have an accuracy of ±5 percent over its operating range.

(Section 114 of the Clean Air Act as amended (42 U.S.C. 7414))

§ 60.374 Test methods and procedures.

(a) Reference methods in Appendix A of this part, except as provided under § 60.8(b), shall be used to determine compliance according to § 60.8 as follows:

(1) Method 12 for the measurement of lead concentrations,

(2) Method 1 for sample and velocity traverses,

(3) Method 2 for velocity and volumetric flow rate,

(4) Method 4 for stack gas moisture, and

(b) For Method 12, the sampling time for each run shall be at least 60 minutes except as provided for in paragraph (c), and the sampling rate shall be at least 0.85 dscm/h (0.53 dscf/min), except that shorter sampling times, when necessitated by process variables or other factors, may be approved by the Administrator.

(c) For grid casting, the sampling time for each Method 12 run shall be at least 180 minutes with a sampling rate of at least 0.85 dscm/h (0.53 dscf/min), except that shorter sampling times, when necessitated by process variables or other factors, may be approved by the Administrator.

(d) When different operations in a three-process operation facility are ducted to separate control devices, the lead emission concentration from the facility shall be determined using the equation:

$$C_{Pb_T} = \sum_{a=1}^{N} C_{Pb_a}(Q_{sd_a}/Q_{sd_T})$$

Where:

C_{Pb_T} is the lead emission concentration for the entire facility.

N is the number of control devices to which separate operations in the facility are ducted.

C_{Pb_a} is the emission concentration from each control device.

Q_{sd_a} is the dry standard volumetric flow rate of the effluent gas stream from each control device.

Q_{sd_T} is the total dry standard volumetric flow rate from all of the control devices.

(e) For lead oxide manufacturing facilities, the average lead feed rate to a facility, expressed in kilograms per hour, shall be determined for each test run as follows:

(1) Calculate the total amount of lead charged to the facility during the run by multiplying the number of lead pigs (ingots) charged during the run by the average mass of a pig in kilograms or by another suitable method.

(2) Divide the total amount of lead charged to the facility during the run by the duration of the run in hours.

(f) Lead emissions from lead oxide manufacturing facilities, expressed in milligrams per kilogram of lead charged, shall be determined using the following equation:

$$E_{Pb} = C_{Pb}Q_{sd}/F$$

Where:

E_{Pb} is the lead emission rate from the facility in milligrams per kilogram of lead charged.

C_{Pb} is the concentration of lead in the exhaust stream in milligrams per dry standard cubic meter as determined according to paragraph (a)(1) of this section.

Q_{sd} is the dry standard volumetric flow rate in dry standard cubic meters per hour as determined according to paragraph (a)(3) of this section.

F is the lead feed rate to the facility in kilograms per hours as determined according to paragraph (e) of this section.

(Section 114 of the Clean Air Act as amended (42 U.S.C. 7414))

2. Appendix A to Part 60 is amended by adding new Reference Method 12 as follows:

Appendix A—Reference Methods

* * * * *

Method 12. Determination of Inorganic Lead Emissions from Stationary Sources

1. Applicability and Principle

1.1 *Applicability.* This method applies to the determination of inorganic lead (Pb) emissions from specified stationary sources only.

1.2 *Principle.* Particulate and gaseous Pb emissions are withdrawn isokinetically from the source and collected on a filter and in dilute nitric acid. The collected samples are digested in acid solution and analyzed by atomic absorption spectrometry using an air acetylene flame.

2. Range, Sensitivity, Precision, and Interferences

2.1 *Range.* For a minimum analytical accuracy of ±10 percent, the lower limit of the range is 100 μg. The upper limit can be considerably extended by dilution.

2.2 *Analytical Sensitivity.* Typical sensitivities for a 1-percent change in absorption (0.0044 absorbance units) are 0.2 and 0.5 μg Pb/ml for the 217.0 and 283.3 nm lines, respectively.

2.3 *Precision.* The within-laboratory precision, as measured by the coefficient of variation ranges from 0.2 to 9.5 percent relative to a run-mean concentration. These values were based on tests conducted at a gray iron foundry, a lead storage battery manufacturing plant, a secondary lead smelter, and a lead recovery furnace of an alkyl lead manufacturing plant. The concentrations encountered during these tests ranged from 0.61 to 123.3 mg Pb/m³.

2.4 *Interferences.* Sample matrix effects may interfere with the analysis for Pb by flame atomic absorption. If this interference is suspected, the analyst may confirm the presence of these matrix effects and frequently eliminate the interference by using the Method of Standard Additions.

High concentrations of copper may interfere with the analysis of Pb at 217.0 nm. This interference can be avoided by analyzing the samples at 283.3 nm.

3. Apparatus

3.1 *Sampling Train.* A schematic of the sampling train is shown in Figure 12–1; it is similar to the Method 5 train. The sampling train consists of the following components:

3.1.1 *Probe Nozzle, Probe Liner, Pitot Tube, Differential Pressure Gauge, Filter Holder, Filter Heating System, Metering System, Barometer, and Gas Density Determination Equipment.* Same as Method 5, Sections 2.1.1 to 2.1.6 and 2.1.8 to 2.1.10, respectively.

3.1.2 *Impingers.* Four impingers connected in series with leak-free ground glass fittings or any similar leak-free noncontaminating fittings. For the first, third, and fourth impingers, use the Greenburg-Smith design, modified by replacing the tip with a 1.3 cm (½ in.) ID glass tube extending to about 1.3 cm (½ in.) from the bottom of the flask. For the second impinger, use the Greenburg-Smith design with the standard tip. Place a thermometer, capable of measuring temperature to within 1°C (2°F) at the outlet of the fourth impinger for monitoring purposes.

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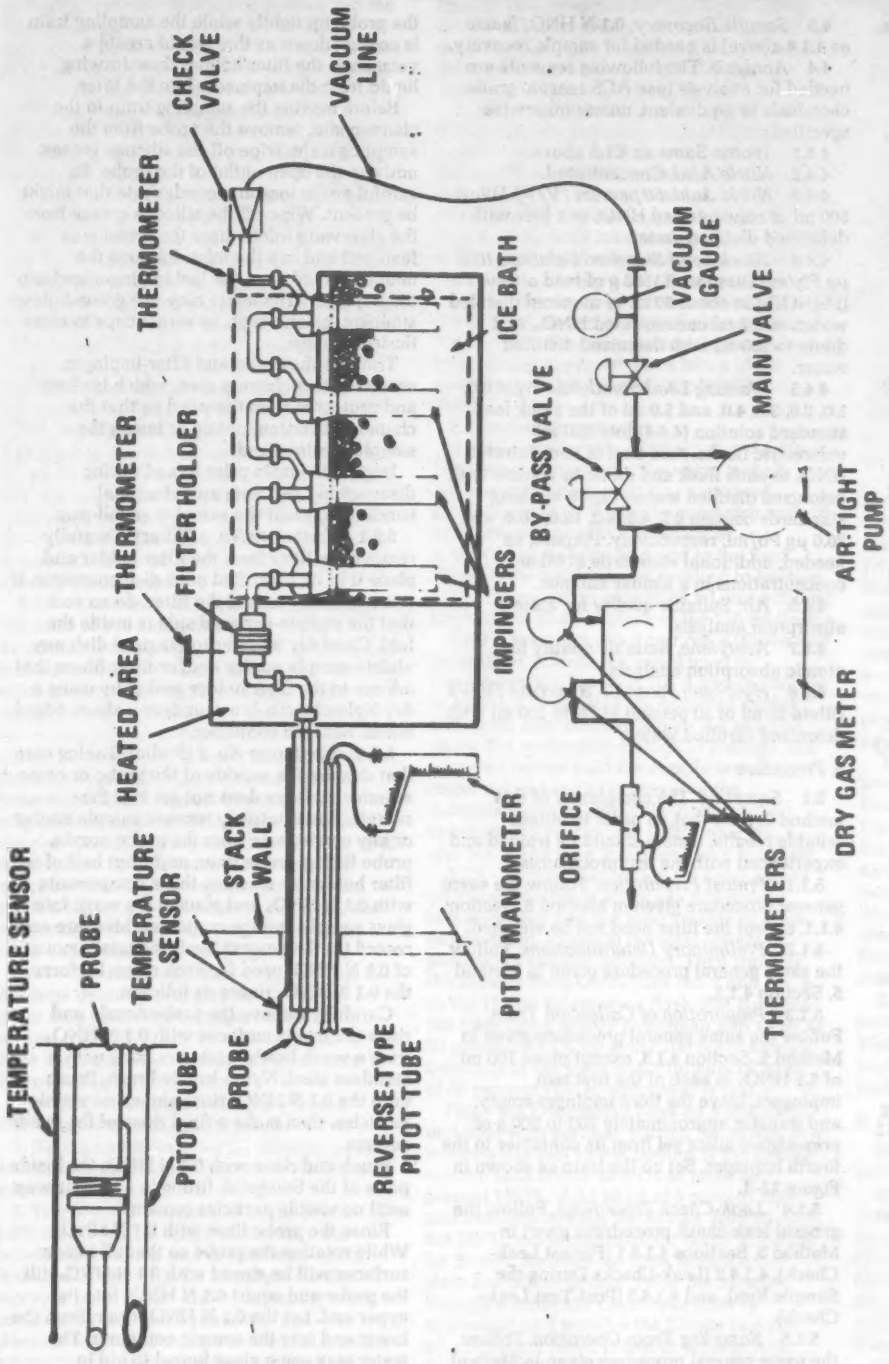

Figure 12-1. Inorganic lead sampling train.

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3.2 *Sample Recovery.* The following items are needed:

3.2.1 *Probe-Liner and Probe-Nozzle Brushes, Petri Dishes, Plastic Storage Containers, and Funnel and Rubber Policeman.* Same as Method 5, Sections 2.2.1, 2.2.4, 2.2.6, and 2.2.7, respectively.

3.2.2 *Wash Bottles.* Glass (2).

3.2.3 *Sample Storage Container.* Chemically resistant, borosilicate glass bottles, for 0.1 N nitric acid (HNO_3) impinger and probe solutions and washes, 1000-ml. Use screw-cap liners that are either rubber-backed Teflon [1] or leak-free and resistant to chemical attack by 0.1 N HNO_3. (Narrow mouth glass bottles have been found to be less prone to leakage.)

3.2.4 *Graduated Cylinder and/or Balance.* To measure condensed water to within 2 ml or 1 g. Use a graduated cylinder that has a minimum capacity of 500 ml, and subdivisions no greater than 5 ml. (Most laboratory balances are capable of weighing to the nearest 0.5 g or less.)

3.2.5 *Funnel.* Glass, to aid in sample recovery.

3.3 Analysis, The following equipment is needed:

3.3.1 *Atomic Absorption Spectrophotometer.* With lead hollow cathode lamp and burner for air/acetylene flame.

3.3.2 Hot Plate.

3.3.3 *Erlenmeyer Flasks.* 1 25-ml, 24/40 $.

3.3.4 *Membrane Filters.* Millipore SCWPO 4700 or equivalent.

3.3.5 *Filtration Apparatus.* Millipore vacuum filtration unit, or equivalent, for use with the above membrane filter.

3.3.6 *Vomumetric Flasks.* 100-ml, 250-ml, and 1000-ml.

4. *Reagents*

4.1 Sampling. The reagents used in sampling are as follows:

4.1.1 Filter. Gelman Spectro Grade, Reeve Angel 934 AH, MSA 1106 BH, all with lot assay for Pb, or other high-purity glass fiber filters, without organic binder, exhibiting at least 99.95 percent efficiency (<0.05 percent penetration) on 0.3 micron dioctyl phthalate smoke particles. Conduct the filter efficiency test using ASTM Standard Method D 2986–71 or use test data from the supplier's quality control program.

4.1.2 *Silica Gel, Crushed Ice, and Stopcock Grease.* Same as Method 5, Section 3.1.2, 3.1.4, and 3.1.5, respectively.

4.1.3 *Water.* Deionized distilled, to conform to ASTM Specification D 1193–74, Type 3. If high concentrations of organic matter are not expected to be present, the analyst may delete the potassium permanganate test for oxidizable organic matter.

4.1.4 *Nitric Acid, 0.1 N. Dilute 6.5 ml of concentrated HNO_3 to liter 1 with deionized distilled water.* (It may be desirable to run blanks before field use to eliminate a high blank on test samples.)

4.2 *Pretest preparation.* 6 HNO_3 is needed. Dilute 390 ml of concentrated HNO_3 to 1 liter with deionized distilled water.

4.3 *Sample Recovery.* 0.1 N HNO_3 (same as 4.1.4 above) is needed for sample recovery.

4.4 *Analysis.* The following reagents are needed for analysis (use ACS reagent grade chemicals or equivalent, unless otherwise specified):

4.4.1 *Water.* Same as 4.1.3 above.

4.4.2 *Nitric Acid.* Concentrated.

4.4.3 *Nitric Acid, 50 percent (V/V).* Dilute 500 ml of concentrated HNO_3 to 1 liter with deionized distilled water.

4.4.4 *Stock Lead Standard Solution, 1000 μg Pb/ml.* Dissolve 0.1598 g of lead nitrate [$Pb(NO_3)_2$] in about 60 ml of dionized distilled water, add 2 ml concentrated HNO_3, and dilute to 100 ml with deionized distilled water.

4.4.5 *Working Lead Standards.* Pipet 0.0, 1.0, 2.0, 3.0, 4.0, and 5.0 ml of the stock lead standard solution (4.4.4) into 250-ml volumetric flasks. Add 5 ml of concentrated HNO_3 to each flask and dilute to volume with deionized distilled water. These working standards contain 0.0, 4.0, 8.0, 12.0, 16.0, and 20.0 μg Pb/ml, respectively. Prepare, as needed, additional standards at other concentrations in a similar manner.

4.4.6 *Air.* Suitable quality for atomic absorption analysis.

4.4.7 *Acetylene.* Suitable quality for atomic absorption analysis.

4.4.8 *Hydrogen Peroxide, 3 percent (V/V).* Dilute 10 ml of 30 percent H_2O_2 to 100 ml with deionized distilled water.

5. Procedure

5.1 *Sampling.* The complexity of this method is such that, in order to obtain reliable results, testers should be trained and experienced with the test procedures.

5.1.1 *Pretest Preparation.* Follow the same general procedure given in Method 5, Section 4.1.1, except the filter need not be weighed.

5.1.2 *Preliminary Determinations.* Follow the same general procedure given in Method 5, Section 4.1.2.

5.1.3 *Preparation of Collection Train.* Follow the same general procedure given in Method 5, Section 4.1.3, except place 100 ml of 0.1 HNO_3 in each of the first two impingers, leave the third impinger empty, and transfer approximately 200 to 300 g of preweighed silica gel from its container to the fourth impinger. Set up the train as shown in Figure 12–1.

5.1.4 *Leak-Check Procedures.* Follow the general leak-check procedures given in Method 5, Sections 4.1.4.1 (Pretest Leak-Check), 4.1.4.2 (Leak-Checks During the Sample Run), and 4.1.4.3 (Post-Test Leak-Check).

5.1.5 *Sampling Train Operation.* Follow the same general procedure given in Method 5, Section 4.1.5. For each run, record the data required on a data sheet such as the one shown in EPA Method 5, Figure 5–2.

5.1.6 *Calculation of Percent Isokinetic.* Same as Method 5, Section 4.1.6.

5.2 *Sample Recovery.* Begin proper cleanup procedure as soon as the probe is removed from the stack at the end of the sampling period.

Allow the probe to cool. When it can be safety handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over it. Do not cap off the probe tip tightly while the sampling train is cooling down as this would create a vacuum in the filter holder, thus drawing liquid from the impingers into the filter.

Before moving the sampling train to the cleanup site, remove the probe from the sampling train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate that might be present. Wipe off the silicone grease from the glassware inlet where the probe was fastened and cap the inlet. Remove the umbilical cord from the last impinger and cap the impinger. The tester may use ground-glass stoppers, plastic caps, or serum caps to close these openings.

Transfer the probe and filter-impinger assembly to a cleanup area, which is clean and protected from the wind so that the chances of contaminating or losing the sample is minimized.

Inspect the train prior to and during disassembly and note any abnormal conditions. Treat the samples as follows:

5.2.1 *Container No. 1* (Filter). Carefully remove the filter from the filter holder and place it in its identified petri dish container. If it is necessary to fold the filter, do so such that the sample-exposed side is inside the fold. Carefully transfer to the petri dish any visible sample matter and/or filter fibers that adhere to the filter holder gasket by using a dry Nylon bristle brush and/or a sharp-edged blade. Seal the container.

5.2.2 *Container No. 2* (Probe). Taking care that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover sample matter or any condensate from the probe nozzle, probe fitting, probe liner, and front half of the filter holder by washing these components with 0.1 N HNO_3 and placing the wash into a glass sample storage container. Measure and record (to the nearest 2-ml) the total amount of 0.1 N HNO_3 used for each rinse. Perform the 0.1 N HNO_3 rinses as follows:

Carefully remove the probe nozzle and rinse the inside surfaces with 0.1 N HNO_3 from a wash bottle while brushing with a stainless steel, Nylon-bristle brush. Brush until the 0.1 N HNO_3 rinse shows no visible particles, then make a final rinse of the inside surface.

Brush and rinse with 0.1 N HNO_3 the inside parts of the Swagelok fitting in a similar way until no visible particles remain.

Rinse the probe liner with 0.1 N HNO_3. While rotating the probe so that all inside surfaces will be rinsed with 0.1 N HNO_3, tilt the probe and squirt 0.1 N HNO_3 into its upper end. Let the 0.1 N HNO_3 drain from the lower end into the sample container. The tester may use a glass funnel to aid in transferring liquid washes to the container. Follow the rinse with a probe brush. Hold the probe in an inclined position, squirt 0.1 N HNO_3 into the upper end of the probe as the probe brush is being pushed with a twisting action through the probe; hold the sample container underneath the lower end of the probe and catch any 0.1 N HNO_3 and sample matter that is brushed from the probe. Run the brush through the probe three times or more until no visible sample matter is carried out with the 0.1 N HNO_3 and none remains on the probe liner on visual inspection. With

[1] Mention of trade names or specific products does not constitute endorsement by the U.S. Environmental Protection Agency.

stainless steel or other metal probes, run the brush through in the above prescribed manner at least six times, since metal probes have small crevices in which sample matter can be entrapped. Rinse the brush with 0.1 N HNO_3 and quantitatively collect these washings in the sample container. After the brushing make a final rinse of the probe as described above.

It is recommended that two people clean the probe to minimize loss of sample. Between sampling runs, keep brushes clean and protected from contamination.

After insuring that all joints are wiped clean of silicone grease, brush and rinse with 0.1 N HNO_3 the inside of the front half of the filter holder. Brush and rinse each surface three times or more, if needed, to remove visible sample matter. Make a final rinse of the brush and filter holder. After all 0.1 N HNO_3 washings and sample matter are collected in the sample container, tighten the lid on the sample container so that the fluid will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether leakage occurs during transport. Label the container to clearly identify its contents.

5.2.3 *Container No. 3* (Silica Gel). Check the color of the indicating silica gel to determine if it has been completely spent and make a notation of its condition. Transfer the silica gel from the fourth impinger to the original container and seal. The tester may use a funnel to pour the silica gel and a rubber policeman to remove the silica gel from the impinger. It is not necessary to remove the small amount of particles that may adhere to the walls and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, the tester may follow procedure for Container No. 3 under Section 5.4 (Analysis).

5.2.4 *Container No. 4* (impingers). Due to the large quantity of liquid involved, the tester may place the impinger solutions in several containers. Clean each of the first three impingers and connecting glassware in the following manner:

1. Wipe the impinger ball joints free of silicone grease and cap the joints.

2. Rotate and agitate each impinger, so that the impinger contents might serve as a rinse solution.

3. Transfer the contents of the impingers to a 500-ml graduated cylinder. Remove the outlet ball joint cap and drain the contents through this opening. Do not separate the impinger parts (inner and outer tubes) while transferring their contents to the cylinder. Measure the liquid volume to within ± 2 ml. Alternatively, determine the weight of the liquid to within ± 0.5 g. Record in the log the volume or weight of the liquid present, along with a notation of any color or film observed in the impinger catch. The liquid volume or weight is needed, along with the silica gel data, to calculate the stack gas moisture content (see Method 5, Figure 5–3).

4. Transfer the contents to Container No. 4.

5. Note: In steps 5 and 6 below, measure and record the total amount of O.1 N HNO_3 used for rinsing. Pour approximately 30 ml of 0.1 N HNO_3 into each of the first three impingers and agitate the impingers. Drain the 0.1 N HNO_3 through the outlet arm of each impinger into Container No. 4. Repeat this operation a second time; inspect the impingers for any abnormal conditions.

6. Wipe the ball joints of the glassware connecting the impingers free of silicone grease and rinse each piece of glassware twice with 0.1 N HNO_3; transfer this rinse into Container No. 4. (*Do not rinse or brush the glass-fritted filter support.*) Mark the height of the fluid level to determine whether leakage occurs during transport. Label the container to clearly identify its contents.

5.2.5 Blanks. Save 200 ml of the 0.1 N HNO_3 used for sampling and cleanup as a blank. Take the solution directly from the bottle being used and place into a glass sample container labeled "0.1 N HNO_3 blank."

5.3 Sample Preparation.

5.3.1 *Container No. 1* (Filter). Cut the filter into strips and transfer the strips and all loose particulate matter into an 125-ml Erlenmeyer flask. Rinse the petri dish with 10 ml of 50 percent HNO_3 to insure a quantitative transfer and add to the flask. (Note: If the total volume required in Section 5.3.3 is expected to exceed 80 ml, use a 250-ml Erlenmeyer flask in place of the 125-ml flask.)

5.3.2 *Containers No. 2 and No. 4* (Probe and Impingers). (Check the liquid level in Containers No. 2 and/or No. 4 and confirm as to whether or not leakage occurred during transport; note observation on the analysis sheet. If a noticeable amount of leakage had occurred, either void the sample or take steps, subject to the approval of the Administrator, to adjust the final results.) Combine the contents of Containers No. 2 and No. 4 and take to dryness on a hot plate.

5.3.3 *Sample Extraction for Lead.* Based on the approximate stack gas particulate concentration and the total volume of stack gas sampled, estimate the total weight of particulate sample collected. Then transfer the residue from Containers No. 2 and No. 4 to the 125-ml Erlenmeyer flask that contains the filter using rubber policeman and 10 ml of 50 percent HNO_3 for every 100 mg of sample collected in the train or a minimum of 30 ml of 50 percent HNO_3, whichever is larger.

Place the Erlenmeyer flask on a hot plate and heat with periodic stirring for 30 min at a temperature just below boiling. If the sample volume falls below 15 ml, add more 50 percent HNO_3. Add 10 ml of 3 percent H_2O_2 and continue heating for 10 min. Add 50 ml of hot (80°C) deionized distilled water and heat for 20 min. Remove the flask from the hot plate and allow to cool. Filter the sample through a Millipore membrane filter or equivalent and transfer the filtrate to a 250-ml volumetric flask. Dilute to volume with deionized distilled water.

5.3.4 *Filter Blank.* Determine a filter blank using two filters from each lot of filters used in the sampling train. Cut each filter into strips and place each filter in a separate 125-ml Erlenmeyer flask. Add 15 ml of 50 percent HNO_3 and treat as described in Section 5.3.3 using 10 ml of 3 percent H_2O_2 and 50 ml of hot, deionized distilled water. Filter and dilute to a total volume of 100 ml using deionized distilled water.

5.3.5 *0.1 N HNO_3 Blank.* Take the entire 200 ml of 0.1 N HNO_3 to dryness on a steam bath, add 15 ml of 50 percent HNO_3, and treat as described in Section 5.3.3 using 10 ml of 3 percent H_2O_2 and 50 ml of hot, deionized distilled water. Dilute to a total volume of 100 ml using deionized distilled water.

5.4 Analysis.

5.4.1 *Lead Determination.* Calibrate the spectrophotometer as described in Section 6.2 and determine the absorbance for each source sample, the filter blank, and 0.1 N HNO_3 blank. Analyze each sample three times in this manner. Make appropriate dilutions, as required, to bring all sample Pb concentrations into the linear absorbance range of the spectrophotometer.

If the Pb concentration of a sample is at the low end of the calibration curve and high accuracy is required, the sample can be taken to dryness on a hot plate and the residue dissolved in the appropriate volume of water to bring it into the optimum range of the calibration curve.

5.4.2 *Mandatory Check for Matrix Effects on the Lead Results.* The analysis for Pb by atomic absorption is sensitive to the chemical composition and to the physical properties (viscosity, pH) of the sample (matrix effects). Since the Pb procedure described here will be applied to many different sources, many sample matrices will be encountered. Thus, check (mandatory) at least one sample from each source using the Method of Additions to ascertain that the chemical composition and physical properties of the sample did not cause erroneous analytical results.

Three acceptable "Method of Additions" procedures are described in the General Procedure Section of the Perkin Elmer Corporation Manual (see Citation 9.1). If the results of the Method of Additions procedure on the source sample do not agree within 5 percent of the value obtained by the conventional atomic absorption analysis, then the tester must reanalyze all samples from the source using the Method of Additions procedure.

5.4.3 *Container No. 3* (Silica Gel). The tester may conduct this step in the field. Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g; record this weight.

6. Calibration

Maintain a laboratory log of all calibrations.

6.1 *Sampling Train Calibration.* Calibrate the sampling train components according to the indicated sections of Method 5: Probe Nozzle (Section 5.1); Pitot Tube (Section 5.2); Metering System (Section 5.3); Probe Heater (Section 5.4); Temperature Gauges (Section 5.5); Leak-Check of the Metering System (Section 5.6); and Barometer (Section 5.7).

6.2 *Spectrophotometer.* Measure the absorbance of the standard solutions using the instrument settings recommended by the spectrophotometer manufacturer. Repeat until good agreement (± 3 percent) is obtained between two consecutive readings. Plot the absorbance (y-axis) versus concentration in μg Pb/ml (x-axis). Draw or compute a straight line through the linear portion of the curve. Do not force the calibration curve through zero, but if the curve does not pass through the origin or at least lie closer to the origin than ± 0.003

absorbance units, check for incorrectly prepared standards and for curvature in the calibration curve.

To determine stability of the calibration curve, run a blank and a standard after every five samples and recalibrate, as necessary.

7. Calculations

7.1 *Dry Gas Volume.* Using the data from this test, calculate $V_{m(std)}$, the total volume of dry gas metered corrected to standard conditions (20 °C and 760 mm Hg), by using Equation 5–1 of Method 5. If necessary, adjust $V_{m(std)}$ for leakages as outlined in Section 6.3 of Method 5. See the field data sheet for the average dry gas meter temperature and average orifice pressure drop.

7.2 *Volume of Water Vapor and Moisture Content.* Using data obtained in this test and Equations 5–2 and 5–3 of Method 5, calculate the volume of water vapor $V_{w(std)}$ and the moisture content B_{ws} of the stack gas.

7.3 *Total Lead in Source Sample.* For each source sample correct the average absorbance for the contribution of the filter blank and the 0.1 N HNO_3 blank. Use the calibration curve and this corrected absorbance to determine the μg Pb concentration in the sample aspirated into the spectrophotometer. Calculate the total Pb content C'_{Pb} (in μg) in the original source sample; correct for all the dilutions that were made to bring the Pb concentration of the sample into the linear range of the spectrophotometer.

7.4 *Lead Concentration.* Calculate the stack gas Pb concentration C_{Pb} in mg/dscm as follows:

$$C_{Pb} = K \frac{C'_{Pb}}{V_{m(std)}} \quad \text{Eq. 12-1}$$

Where:

K = 0.001 mg/μg for metric units.
= 2.205 lb/μg for English units.

7.5 *Isokinetic Variation and Acceptable Results.* Same as Method 5, Sections 6.11 and 6.12, respectively. To calculate v_s, the average stack gas velocity, use Equation 2–9 of Method 2 and the data from this field test.

8. Alternative Test Methods for Inorganic Lead

8.1 *Simultaneous Determination of Particulate and Lead Emissions.* The tester may use Method 5 to simultaneously determine Pb provided that (1) he uses 0.1 N HNO_3 in the impingers, (2) he uses a glass fiber filter with a low Pb background, and (3) he treats and analyzes the entire train contents, including the impingers, for Pb as described in Section 5 of this method.

8.2 *Filter Location.* The tester may use a filter between the third and fourth impinger provided that he includes the filter in the analysis for Pb.

8.3 *In-stack Filter.* The tester may use an in-stack filter provided that (1) he uses a glass-lined probe and at least two impingers, each containing 100 ml of 0.1 N HNO_3, after the in-stack filter and (2) he recovers and analyzes the probe and impinger contents for Pb.

9. Bibliography

9.1 *Perkin Elmer Corporation.* Analytical Methods for Atomic Absorption Spectrophotometry. Norwalk, Connecticut. September 1976.

9.2 *American Society for Testing and Materials.* Annual Book of ASTM Standards. Part 31; Water, Atmospheric Analysis. Philadelphia, Pa. 1974. p. 40–42.

9.3 *Klein, R. and C. Hach.* Standard Additions—Uses and Limitations in Spectrophotometric Analysis. *Amer. Lab.* 9:21–27. 1977.

9.4 *Mitchell, W. J. and M. R. Midgett.* Determining Inorganic and Alkyl Lead Emissions from Stationary Sources. U.S. Environmental Protection Agency, Emission Monitoring and Support Laboratory. Research Triangle Park, NC. (Presented at National APCA Meeting. Houston. June 26, 1978.)

9.5 Same as Method 5, Citations 2 to 5 and 7 of Section 7.

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(Sections 111, 114, and 301(a) of the Clean Air Act as amended (42 U.S.C. 7411, 7414, and 7601(a)))

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